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② **LEVEL II**

CHARACTERIZATION OF TUNGSTEN DISPENSER CATHODES USING ISS AND SIMS

*MECHANICS AND SURFACE INTERACTIONS BRANCH
NONMETALLIC MATERIALS DIVISION*

OCTOBER 1979

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Final Report for period January 1979 - June 1979

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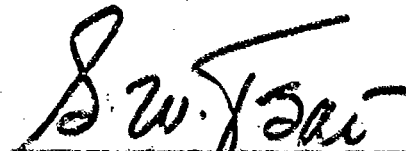
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Ion Scattering Spectrometry (ISS) and Secondary Ion Mass Spectrometry (SIMS) are used to characterize surface chemistry of barium-activated tungsten dispenser cathodes. Experiments were carried out at room temperature and at temperatures in which electron emission was occurring from the cathode structures. It was found that when the cathode structure was heated to a point at which electron emission began, barium concentration at the surface also increased. At that temperature, the barium is		

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generated, transported through the porous tungsten matrix, and spread over the emitting tungsten surface. Apparently, sputtering rates from these barium rich surfaces were very high and when ion beams were used at conventional energies (1000 volts or more), the sputtering rate was sufficiently large to suggest that incomplete coverage of barium existed. When very low ion beam voltages were used (in the neighborhood of 200-300 volts), and beam rastering techniques were employed, then results showed that the surfaces following activation were completely covered by barium. Under these conditions of complete barium coverage, no oxygen was observed. When the surface was sputtered and tungsten appeared, oxygen also increased, but not to a point to indicate a full oxide coating. A model of Ba bonded to O bonded to W is suggested.

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FOREWORD

This technical report was prepared by W. L. Baun, Mechanics and Surface Interactions Branch, Nonmetallic Materials Division, Air Force Materials Laboratory (AFML/MBM). This work was initiated under Project 2303, "Chemistry Research," Task 2303Q1 "Surface Chemistry," and W. U. D. #41 "Analysis of Traveling Wave Tubes." The work Unit monitor is Capt. Bruce Lamartine.

This report covers work conducted inhouse during the period January 1979 to June 1979.

Extremely useful discussions with T. W. Haas concerning cathode construction, operation, and background are gratefully acknowledged. Bruce Lamartine is thanked for supplying the cathode structures and providing technical information about the cathodes. Dolph Biermann is thanked for the careful work he performed in making electrical connections to the cathodes.

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SECTION I INTRODUCTION

The oxides of the alkaline earth elements (barium, strontium, and calcium) have been the key ingredients in the manufacturing of oxide and dispenser cathodes for many years and are currently the most widely used emissive surfaces in commercial cathodes. Types of cathodes, methods of fabrication, and theory of operation have been summarized by Haas and co-workers.¹ The mechanism of operation of barium-activated tungsten impregnated dispenser type cathodes, as shown in Figure 1, has been thoroughly investigated by numerous authors, some accounts published over 20 years ago.²⁻⁴ This cathode features a porous tungsten cap impregnated with a mixture of alkaline earth oxides or carbonates. When this mixture is heated to the neighborhood of 1000°C, decomposition occurs, and a reaction takes place between barium oxide and tungsten to produce barium metal. The free barium diffuses through the pores of the tungsten cap by migration over the pore walls and flow of the vapor. It is thought that the barium on reaching the surface spreads from each pore, and the surface is covered by a monolayer of barium bonded to oxygen. In the cathodes used in this work, there is not a separate reservoir, but rather, the activating mixture is impregnated in the porous tungsten. Work by Forman,⁵ based on Auger spectroscopy and desorption measurements on impregnated cathode surfaces results in his conclusion that barium coverage may not be complete and the coverage decreases with time. Very recent results published by Forman⁶ elucidate some uncertainties present in the earlier model.

It is the purpose of this report to review ion scattering and secondary ion mass spectrometry measurements made on barium-activated tungsten impregnated dispenser type cathodes both at room temperature and at temperatures in which electron emission was occurring.

Ion scattering spectrometry is an ideal tool to use for investigating small changes in chemistry taking place at the surface. It is sensitive only to the outermost layers of atoms at the surface. The use of ion scattering is a relatively recent development. Rubin⁷ applied MeV ion scattering to analyze the surface of metals in 1959. Panin⁸ and Ball et al.⁹ investigated the intermediate energy range and Smith^{10,11} used low energy (about 1 KeV) noble gas ions to probe the surface of a variety of materials. Smith^{10,11} found that when the energy of the ions was lowered, the scattered ion spectrum became simpler and sharper and approached the behavior expected on the basis of a binary scattering event from a single surface atom. Therefore, the energy E_1 retained by an ion of mass M_{ion} , which with an original energy E_0 , after scattering from an atom of mass M_{atom} , through an angle, theta, is given by Equation 1 which is based on the conservation of kinetic energy and momentum.

$$\frac{E_1}{E_0} = \frac{M_{ion}^2}{M_{ion} + M_{atom}} \left\{ \cos\theta + \left(\frac{M_{atom}^2}{M_{ion}^2} - \sin^2\theta \right)^{1/2} \right\}^2 \quad (1)$$

For 90° scattering which is frequently used this reduces to a very simple relationship:

$$E_1/E_0 = (M_{atom} - M_{ion}) / (M_{atom} + M_{ion}). \quad (2)$$

An inherent feature of ion scattering which may be considered an advantage or disadvantage is the simultaneous sputtering of the atoms from the surface as energy is transferred to the surface from the ion beam. It is an advantage in that the concentration of the various atomic species may be followed with depth. On the

other hand, it is a disadvantage because damage is being produced by the sputtering. Once the atom sputters from the surface, the sample is changed, and the exact experiment on that spot may not be repeated. The sputtering and ionization process then forms the basis of secondary ion mass spectrometry. An excellent review by Winters¹² discusses experiment and theory of sputtering.

SECTION II

EXPERIMENTAL

A commercial ion scattering spectrometer, using a cylindrical mirror analyzer (CMA) was used in this work. The cylindrical mirror analyzer with a co-axial small beam ion gun, permits acceptance of ions scattered at 137.7 degrees throughout a complete 360 degree solid angle about the incident primary beam. Figure 2A schematically indicates physical arrangement of gun, sample, and analyzer. Figure 2B shows a typical ion scattering spectrum of ^3He scattering from aluminum oxide. Note that the spectrum is extremely simple, consisting only of one peak for oxygen and one peak for aluminum. A vacuum chamber was ion and sublimation pumped into the mid 10^{-9} Torr range, and backfilled with helium, neon, or argon to about 3×10^{-5} Torr. The sample was not biased. Target current therefore represents the sum of arriving positive ions and departing secondary electrons. This same target current measuring circuit was also used to measure the point at which electron emission began from the specimen. The polarity of the current measuring circuit was reversed for electron measurement.

The mini-beam ion gun system was used and is based on a three-lens system that permits a convenient focusing from high intensity small diameter beams to large diameter focused beams. The smallest ion beam spot capable of resolving 100 micrometers can be scanned or rastered over an area greater than 100×100 diameters when an image of a sample is desired. The rastered area is a function of the target distance as well as the beam voltage, and for a target distance of one inch and a beam energy of 1 KeV, about 1 cm^2 can be scanned. Cathode structures were mounted on a manipulator capable of providing x, y, and z motion. Connections to the cathode structure for applying dc power were

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spot welded and connection was made to the cathode structure itself to allow ion beam current measurements as well as electron emission measurements as mentioned above.

SECTION III

DISCUSSION AND RESULTS

Initial ISS data using ^4He at 1500 volts showed the presence of oxygen and tungsten on the surface and indications of unresolved elements in the aluminum-silicon region and in the potassium-calcium region. The scattering from barium was observed as a low energy shoulder on the tungsten scattering peak. Initial scans of SIMS spectra showed sodium, aluminum, potassium, and calcium present on the surface along with small amounts of hydrocarbons. Both sodium at mass 23 and aluminum at mass 27 were accompanied by mass +1 peaks which perhaps are the hydride species associated with an active surface which has been exposed to water vapor. Ion scattering spectra were then obtained with $^{20}\text{Ne}^+$ at 1500 volts, and both barium and tungsten were observed on the surface. With continued sputtering, the barium was reduced to a low level as seen in Figure 3 which apparently represents Ba at grain boundaries. The spectrum taken then with $^4\text{He}^+$ showed that the oxygen was greatly reduced and the other elements were virtually removed from the surface of the tungsten. Unfortunately, in the $^4\text{He}^+$ data, elements such as tungsten exhibit spectra in which the scattering processes are not all of the binary elastic collision type. Many collisions occur which produce varying amounts of energy loss at the surface and below the surface producing a long tail to the low energy side of the elastic peak. The chamber was again back-filled with ^{20}Ne and activation experiments were begun. Current was passed through the heater (which was potted in the assembly containing the cathode) in increments which could be related to the cathode temperature determined from the curve obtained by the use of a thermocouple in earlier experiments and by optical pyrometer measurements at the higher end of the current range. During early heating, ion scattering spectra showed only barium and tungsten in the ratio obtained from the original surface of the

cathode. Positive SIMS in the mass range 0-70 amu showed only the presence of hydrocarbons, small traces of aluminum and calcium. Sodium and potassium levels were very low at this point. Both ISS and SIMS data were obtained at 0.1 amp intervals between zero and one ampere heater current. At 0.5 (between 400-500°C) ampere there was very little change in the ion scattering data but a very large increase in SIMS peaks was indicated. Peaks due to hydrocarbons in the CH_n , C_2H_n , and C_3H_n range were observed in abundance. Potassium and sodium ions also increased during this same period of heating. At 0.8 (about 800°C) ampere it appeared that there were some small changes taking place in the ratio of the scattered peaks barium/tungsten. To determine if there was a diffusion over a long period of time at this current level of the barium to the surface, the sample was soaked for one hour at this temperature and the spectrum shown in Figure 4 was obtained. The ratio of barium to tungsten is somewhat increased in this spectrum and shows an even larger increase if a large area is rastered or if the beam is spread over a larger area and a lower ion beam voltage is used. When activation temperatures are reached and electron emission has begun, the scattered intensity from tungsten drops dramatically, suggesting that it is being obscured by surface atoms. The barium sputtering rate, especially with neon, appears to be very high, and it seems quite difficult to obtain a spectrum of the original coverage of barium on the tungsten surface. Even with helium at 1500 volts the barium was rapidly removed from the surface, particularly when the specimen was at activation temperatures. In an effort to determine if complete coverage of the surface by barium was taking place, spectra with neon were obtained at voltages as low as 200 volts. Measurements made very rapidly, first at the barium position and then at the tungsten position, showed that complete coverage of barium on the surface was obtained. However, even at low voltages, the time taken to obtain a complete trace, that is, 30 seconds to

one minute always resulted in the appearance of a small amount of tungsten present on the surface. A trace taken with $^{20}\text{Ne}^+$ at 300 volts primary ion energy is seen in Figure 5. The tungsten, under these experimental conditions, seemed to be somewhat shifted from the peak obtained from the pure, clean tungsten surface. This may indicate that the neon ions are being scattered by the tungsten from just below the surface rather than on the first monomolecular layer. These experiments show that activation does take place at current settings of 0.8A. and above and that during activation at lower temperatures, impurities diffuse to the surface. These impurities include hydrocarbons and sodium and potassium.

Positive secondary ion mass spectra obtained during the process of activation also showed the above mentioned impurities. Some of these spectra, as seen in Figure 6, illustrate the changes that take place in the cathode surface upon heating and subsequent electron emission. The spectra indicate that at low temperatures (Fig. 6A) before electron emission begins that the surface is covered primarily with small amounts of hydrocarbons, sodium, potassium and calcium, apparently from the CaO additive. In Figure 6B with an increase in current to 0.7 of an ampere flowing through the heater, the spectrum changes dramatically. The hydrocarbons, such as C, CH, CH_2 , CH_3 , and C_2H_4 increase greatly along with both sodium and potassium. Calcium remains at a high level. This is perhaps the temperature at which the species present as a carbonate or as organic impurities begin to decompose. In Figure 6C, where the sample has been heated to the point at which electron emission has begun, even further changes are observed. Now the potassium at masses 39 and 41 have increased tremendously, perhaps on the order of 10^3 to 10^4 . In addition, a low energy tail on the mass 39 peak is observed and is always observed on spectra taken after activation and during electron emission. This

phenomenon may suggest sputtering of vapor species which are not actually bound on the surface and may occur as neutrals or ions are sputtered from the surface as they emerge from the grain boundaries. At this same temperature at higher masses, the barium is also observed. The barium spectra show similar kind of low energy tailing as seen in the potassium. Such spectra taken during electron emission are shown in Figure 7 in Ba^+ and BaO^+ regions. The small intensity of BaO^+ as compared to Ba^+ would suggest that the barium is not present as an oxide. The spectrum effects such as the low energy tailing may give important insights into the behavior of the cathode at temperatures at which electron emission is occurring. Other changes that are taking place such as the large decrease in the Ne^{++} line suggest that large electrical differences occur as the sample is heated and with the ejection of electrons from the surface, completely different ion neutralization phenomena may take place.

Similar activation experiments were carried out on a second cathode. These experiments gave essentially identical results except that electron emission and an increase in barium concentration at the surface began at lower current settings between 0.6 ampere and 0.7 ampere. Actual cathode temperature may be approximately the same as in the original experiment but, because of the somewhat different geometry and absence of potting of the heater in this structure, it may require less power for the same cathode temperature. Exact cathode temperatures and activation temperatures were not emphasized in this work. Rather, it was desired to know surface composition changes during and after activation. The cathode was sputtered clean, heated, and reactivated numerous times during this set of experiments. Scattered ions from a rastered primary beam were also used in this phase of the work to produce a picture of the surface elemental distribution. Several ion beam compositions were used including $^4He^+$,

$^{20}\text{Ne}^+$, $^{40}\text{Ar}^+$, and a combination of $^{20}\text{Ne}^+$ and $^4\text{He}^+$. The mixture of $^{20}\text{Ne}^+$ and $^4\text{He}^+$ proved to be useful for using the neon scattering peaks to resolve the barium and tungsten and at the same time for following the oxygen concentration using the scattered helium peaks. The use of neon and helium is illustrated in Figure 8 where A shows the spectrum obtained from the clean, unactivated tungsten structure of the cathode. Compared with this then is the spectrum obtained with the cathode cold following activation and replenishing of the surface with barium. Under these circumstances, oxygen is always present, and sometimes a trace of carbon is observed. Apparently barium is an excellent getter and quickly forms a partial monolayer at the surface of barium oxide. To show that this was just a partial monolayer, an experiment was performed in which the surface was covered with a film of barium and then exposed to a partial pressure of air in the system for a short time. This surface is shown to contain much more oxygen than the activated surfaces as seen in Figure 9 and probably represents the formation of a full monolayer of barium oxide.

Rastering and specimen imaging was employed using this cathode. Rastering of the beam over a relatively large area reduces the sputtering rate and allows obtaining an average composition over the entire surface. In addition, the cylindrical mirror analyzer will accept scattered ions over a relatively large area allowing ion imaging of the surface during the rastering. Figure 10A shows a barium scattered image taken on a room temperature cathode after it was activated and then sputtered with the medium spot size using $^{20}\text{Ne}^+$ near the center of the cathode. The dark spot in the center is tungsten and indicates a spot cleaned of barium. In Figure 10B then the same surface is again imaged, but now after temperature has been reached where emission has begun. As can be seen, the entire surface has "healed itself" and is covered with barium. Some interesting patterns were observed at times in both the barium and the tungsten images which may suggest that some

inhomogeneous regions of barium emission are obtained. Long time sputtering at high energies seemed to create spots which were damaged or implanted with He or Ne ions and caused inhomogeneous Ba coverage upon activation. As in the previous cathode structure it was observed here that the emission of electrons at temperature from this cathode structure was accompanied by the formation of a monolayer of barium on the surface. There are strong indications that this monolayer of barium present during electron emission is in the metallic state. Crude oxidation studies of the activated film showed extremely rapid oxidation and a large increase of scattering from oxygen and in addition positive SIMS spectra taken in the Ba^+ and BaO^+ region during electron emission showed an extremely small abundance of BaO^+ formed. BaO^+ intensities with respect to Ba^+ increased on oxidized surfaces.

SECTION IV

CONCLUSIONS

This work using ion scattering and secondary ion mass spectrometry on barium-activated tungsten impregnated dispenser type cathodes indicates the activation procedure causes complete coverage of the tungsten surface by the barium atoms. The association of small amounts of oxygen when barium is sputtered and tungsten appears may suggest either a gettering effect of the small amount of oxygen in the system, or it may indicate scattering from oxygen atoms supplied by the tungsten to which the barium is bonded at the first monolayer. More recent experiments favor the latter mechanism and will be reported separately along with results on the detailed analysis of the low energy sputtered ions.¹³ The present results suggest the activated cathode has a full monolayer of metallic barium adsorbed on a monolayer or less of oxygen on tungsten. The emission of alkali elements upon heating should be investigated further to determine origin, length of time released, effect on work function, final resting place, and effect on cathode or TWT life.

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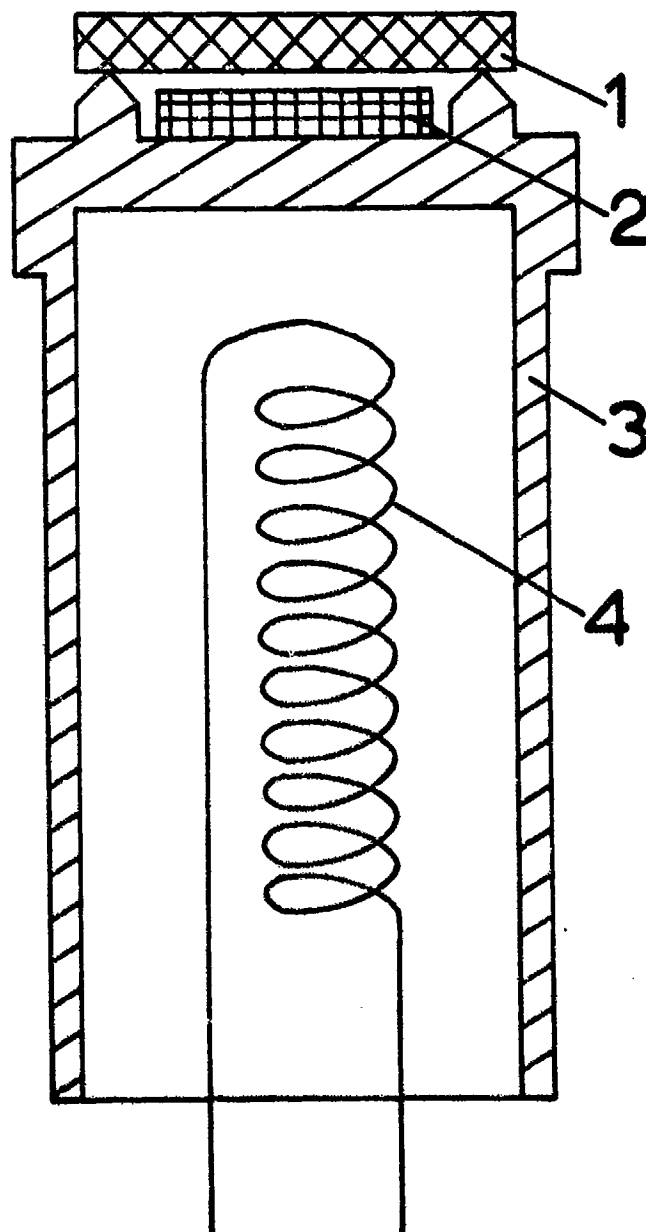


Figure 1. Basic Construction of a Dispenser Cathode. 1. Porous tungsten, 2. Chamber containing reserve of alkaline earth oxide or carbonate with other ingredients, 3. Molybdenum cylinder, 4. Heater filament (cathodes used here used impregnated activator, rather than separate reservoir).

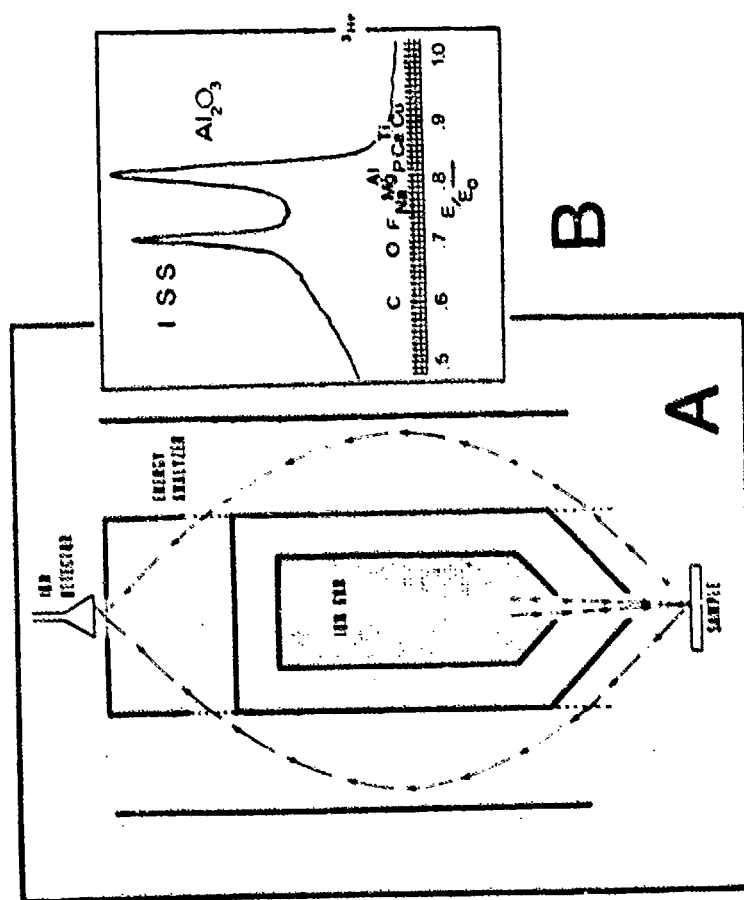


Figure 2. A. Schematic of the Ion Scattering Spectrometry Experiment.
 B. A Typical Ion Scattering Spectrum ${}^3\text{He}^+ \rightarrow \text{Al}_2\text{O}_3$ Showing Simplicity of Spectrum.

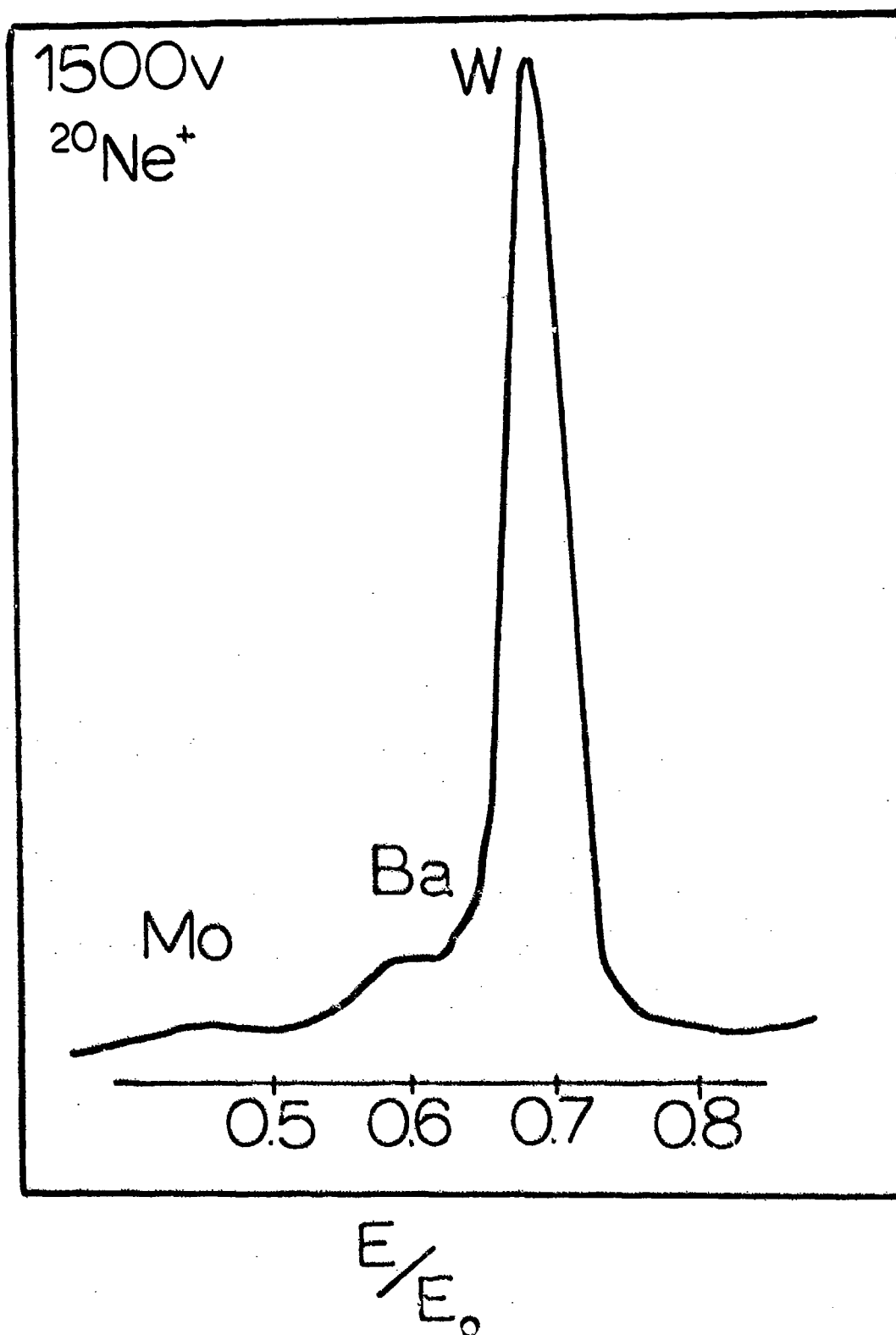


Figure 3. Ion Scattering Spectrum from Sputtered Clean Cathode $^{20}\text{Ne}^+$ at 1500V.

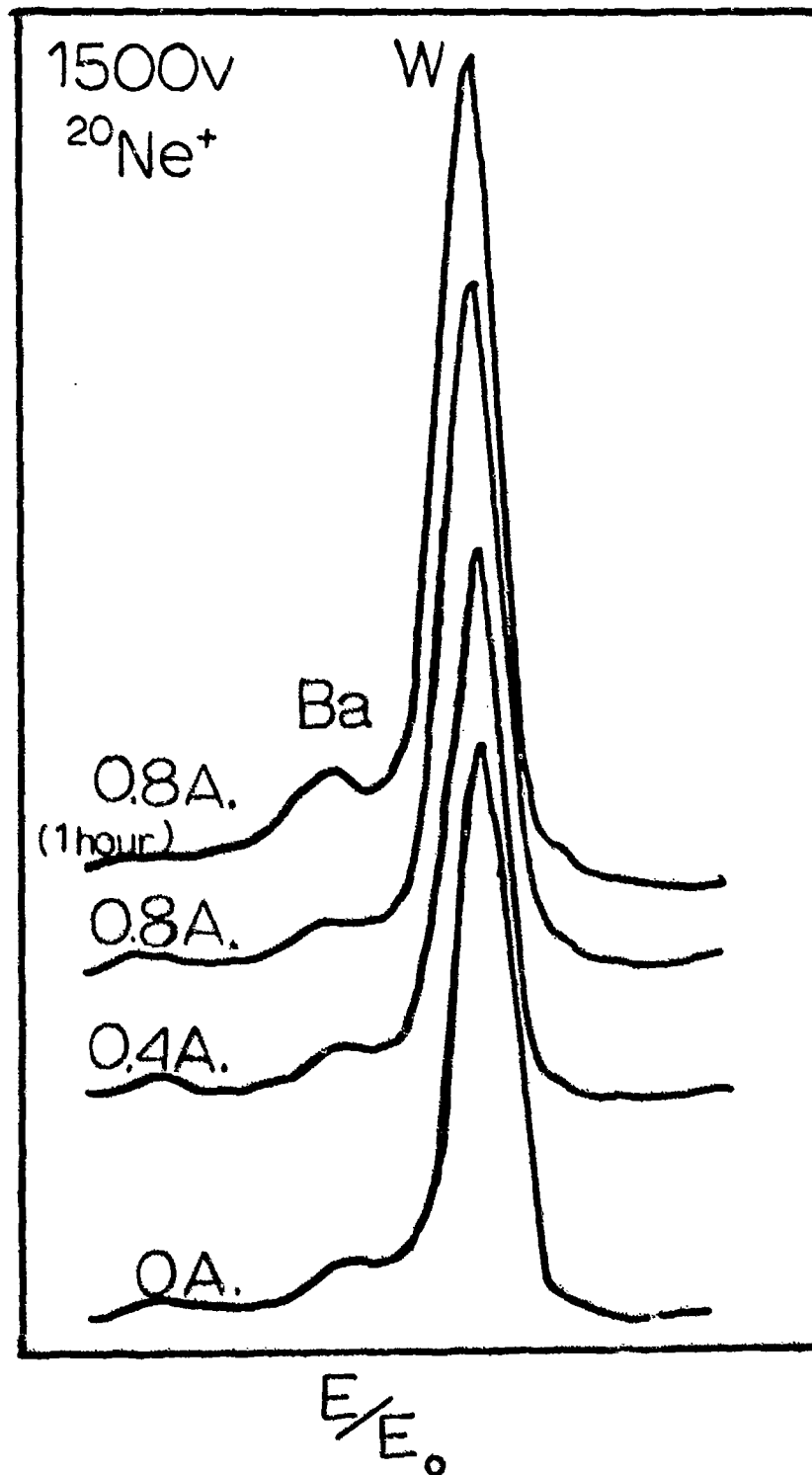


Figure 4. Ion Scattering Spectrum from Impregnated Dispenser Cathode at Room Temperature (0A) and During Heating.

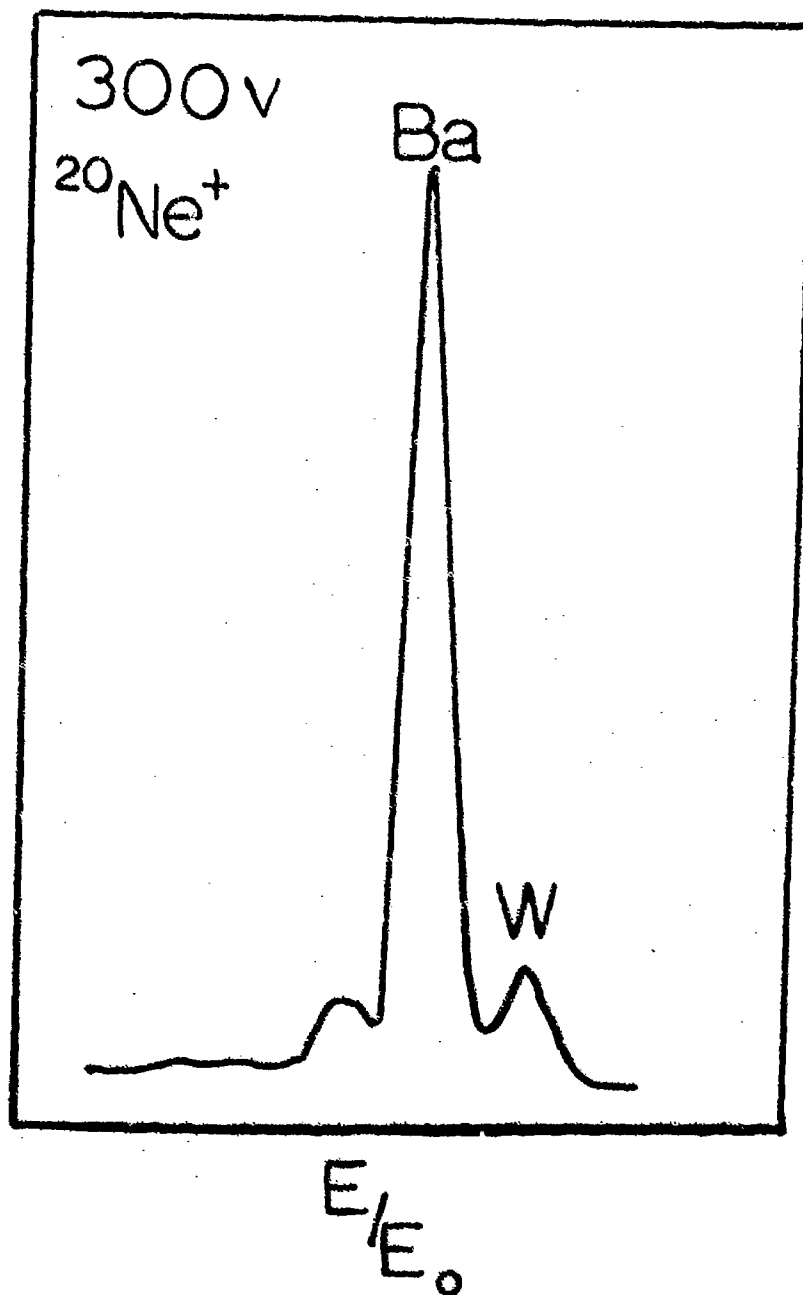


Figure 5. Ion Scattering Spectrum Taken with Low Energy $^{20}\text{Ne}^+$ of Tungsten Impregnated Dispenser Cathode Heated to Cause Electron Emission.

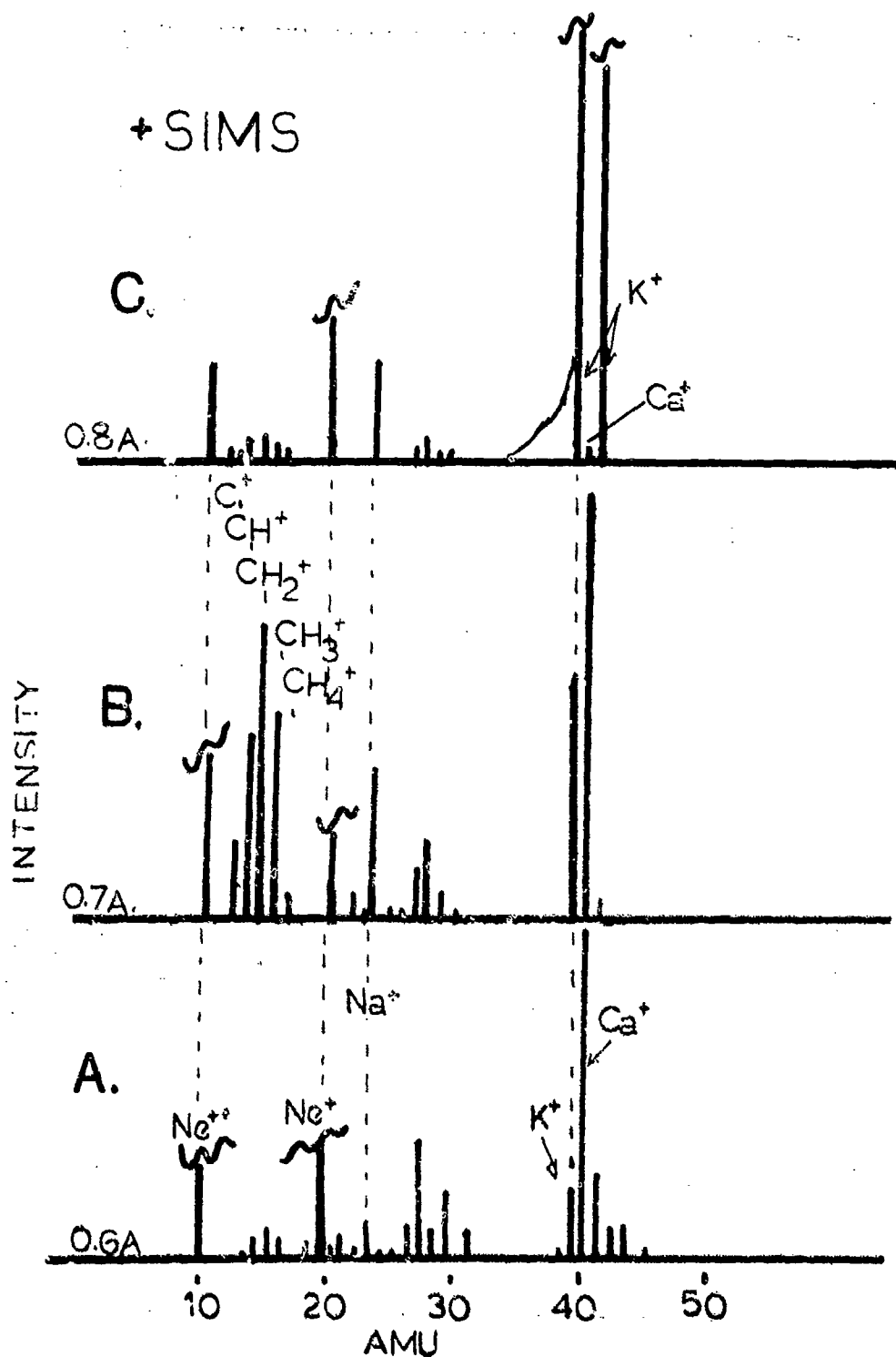
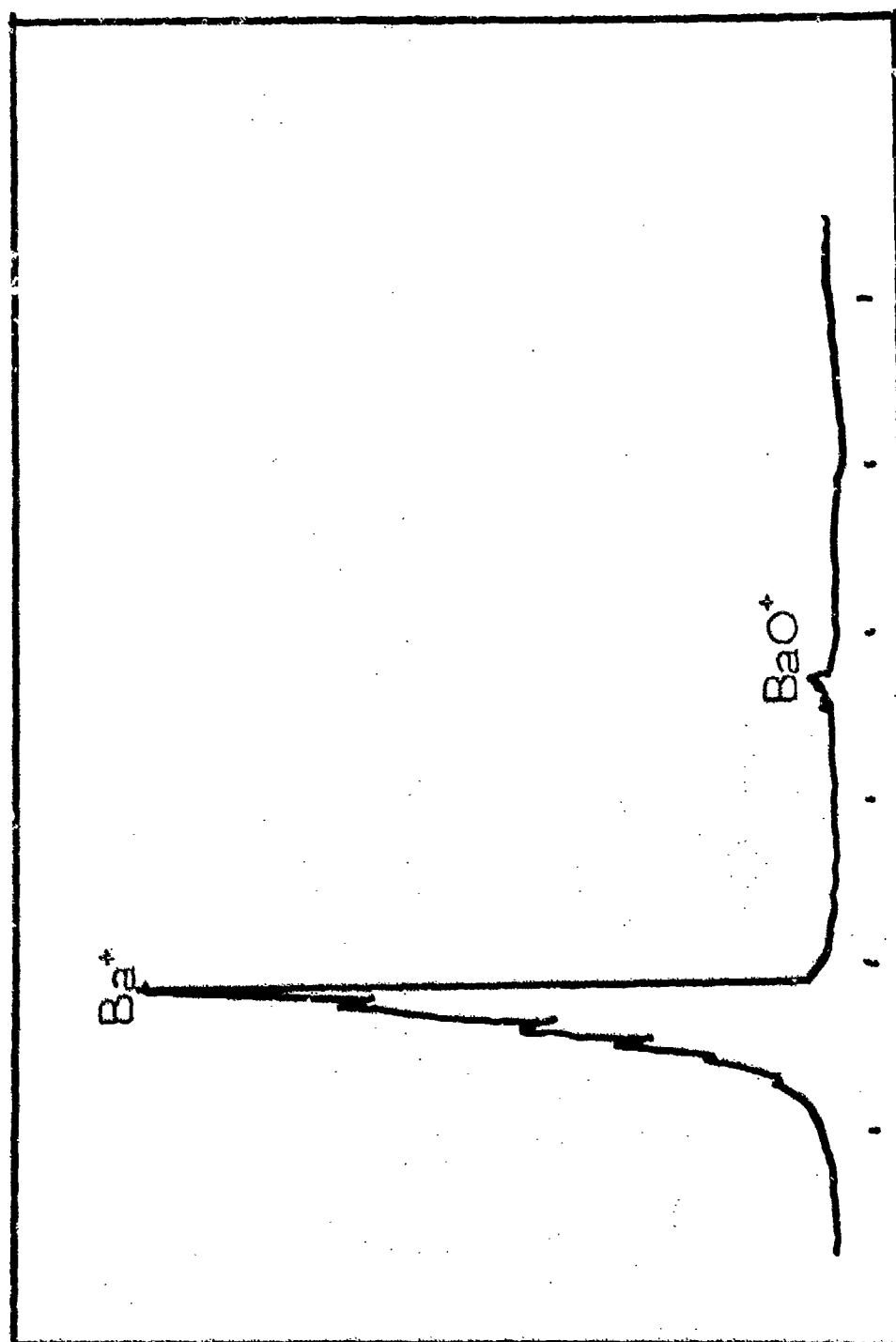


Figure 6. Secondary Ion Mass Spectra from Impregnated Tungsten Cathode.
 A. +SIMS Data During Low Temperature Heating (0.6A heater current).
 B. +SIMS Data Just Before Electron Emission Begins (0.7A heater current).
 C. +SIMS Data When Electron Emission Begins (0.8A heater current).



ATOMIC MASS

Figure 7. Positive Secondary Ion Mass Spectrum of a Hot Immregnated Tungsten Cathode Using $^{20}Ne^+$ at 1500V.

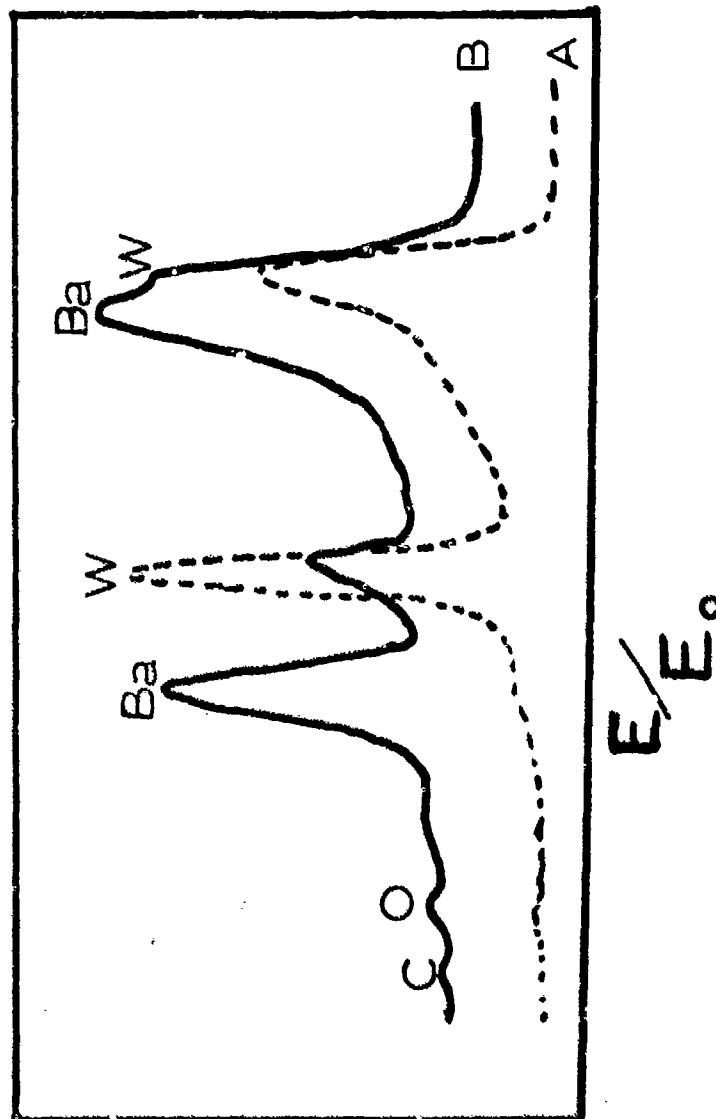


Figure 8. Ion Scattering Spectra from Cathode Structure Using ${}^4\text{He}^+ + {}^{20}\text{Ne}^+$.
 A. Sputtered Clean, Unheated Cathode. B. Activated Cathode Following Cooling.

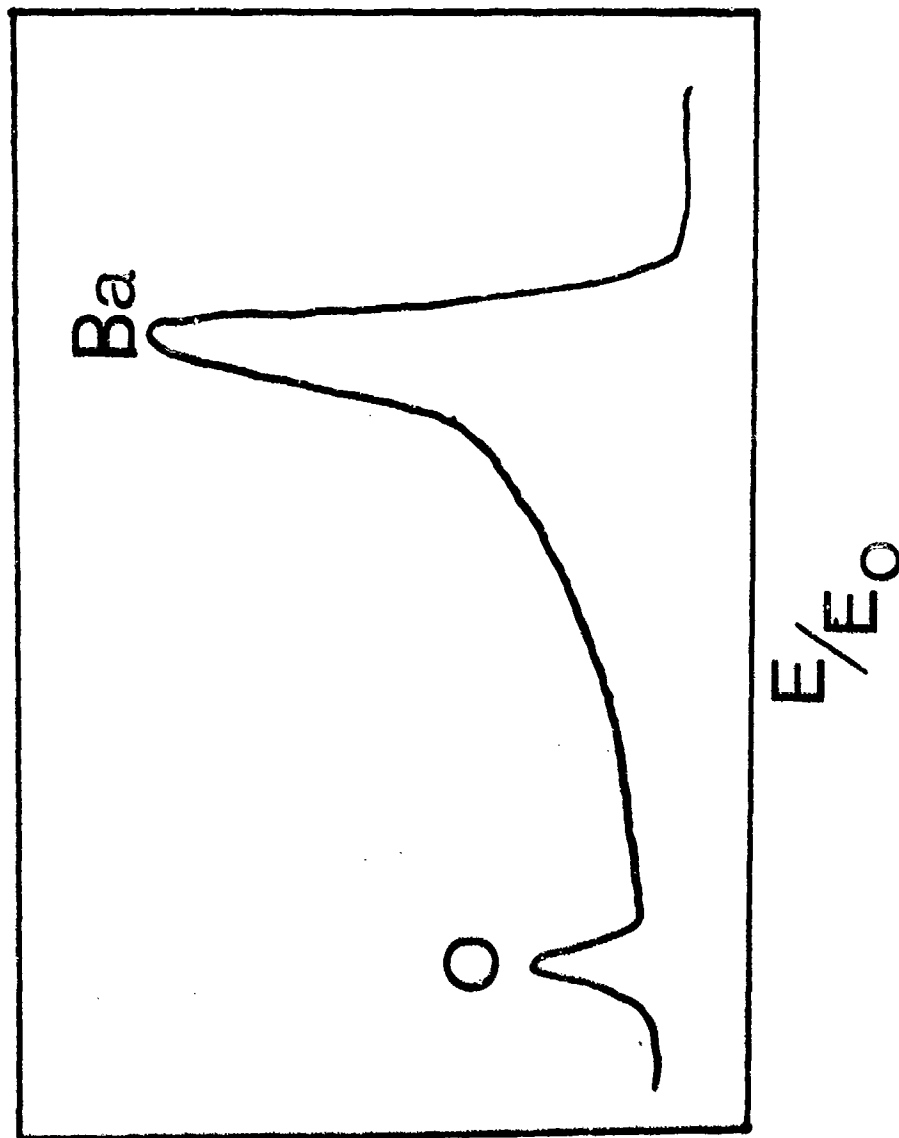
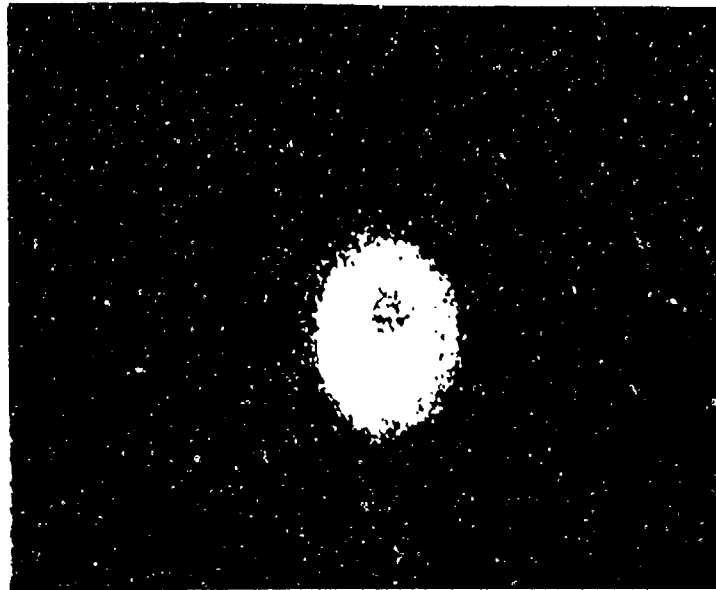


Figure 9. Ion Scattering Spectrum from Unheated Cathode Structure Following Activation and Exposure of Activated Surfaces to a Partial Pressure of Air.



a



b

Figure 10. Scattered Ion Image from Ba: (a) On Room Temperature Cathode after Activation and Ne^+ Bombardment with Medium Spot Near Center of Cathode, (b) Following Reactivation Showing Immediate Ba Coverage.